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THE DETERMINATION OF DISSOLVED SOLIDS

Dissolved solids refer to the quantity of material that remains as a residue after the water is removed from a filtered portion of sample by evaporation. The gravimetric test does not determine specific chemical substances - only a general class which have a similar response under the same test conditions. By assuming the dissolved solids content arises solely from ionic species, this parameter can be estimated by calculating the contribution of ten common ions or from the conductivity provided the latter is less than 450 micromhos/cm.

Man's activities tend to steadily increase the dissolved solids content of our water resources both by the production of wastes and by chemical treatments designed to remove harmful substances. Dissolved solids may render a water unsuitable as a source for drinking water, agricultural use or industrial activity. The permissible criteria for a public surface water source or irrigation is 500 mg/l; source limits for some industries are 100 mg/l (textiles), 500 mg/l (food processing) and 750 mg/l (petroleum refining). Due to potentially harmful osmotic effects on aquatic species and wildlife, the dissolved solids concentration of effluents should not increase the background level of the receiving body by more than one third.

1. Sample Handling and Preservation

Samples should be collected in glass bottles and refrigerated. Preservatives must not be added.

2. Selection of Method

Three methods of determining dissolved solids are available, but the gravimetric is the most commonly employed.

- a) Gravimetric (evaporation of a 50 ml aliquot of filtrate) is applied to all industrial and sewage samples and to all samples with a conductivity in excess of 450 micromhos/cm. It is applied to some samples with conductivities ranging up to 450 micromhos/cm.
- b) Dissolved solids are estimated from the conductivity if requested and if the conductivity is less than 450 micromhos/cm.

- c) Although never reported, dissolved solids are estimated by the laboratory staff from the measured values for *ten* parameters whenever the appropriate data is available; this calculated estimate is utilized as a quality control check.

DISSOLVED SOLIDS
GRAVIMETRIC - METHOD A

SUMMARY

Substance determined.	The residue from a filtered aliquot of sample which has been evaporated at $103 \pm 2^{\circ}\text{C}$.
Interpretation of results.	The dissolved solids, which are expressed in mg/l , include ionic species, colloidal matter, and nonionic substances. Volatile constituents are partially recovered at best.
Principle of method.	A volume of sample is vacuum filtered through pre-washed fibre glass filter paper; a 50 ml aliquot of filtrate is evaporated overnight at $103 \pm 2^{\circ}\text{C}$, and the dissolved solids calculated in mg/l from the weight of residue.
Time required for analysis.	2 days.
Range of application.	Any sample which is filterable under the specified experimental conditions.
Standard deviation.	3.94 mg/l for rivers and lakes in the range 0 - 350 mg/l . 12.7 mg/l for sewage and trade wastes in the range 0 - 500 mg/l .
Accuracy	Not applicable.
Limit of detection.	3.29s \equiv 15.5 mg/l (Rivers and lakes) 41.8 mg/l (Sewages and trade wastes)
Interferences and shortcomings.	Some samples are not amenable to filtration while others contain oily substances or acids that inhibit evaporating to a constant weight; volatile constituents are incompletely recovered at best.
Minimum volume of sample.	75 ml.
Preservation and sample container.	Glass bottles are acceptable, and samples should be refrigerated. Preservatives must not be added.

DISSOLVED SOLIDS
GRAVIMETRIC METHOD

1. Introduction

A portion of sample is vacuum filtered through pre-washed Reeve Angel 934AH glass fibre filter discs; a 50 ml aliquot of filtrate is evaporated overnight at $103 \pm 2^{\circ}\text{C}$. Weighings are performed on balances with 5-decimal place resolution.

2. Interferences and Shortcomings

Some samples cannot be filtered under the specified experimental conditions. Volatile constituents are lost during the evaporation step while the presence of oils, greases, and acids inhibits evaporation to a constant weight.

3. Apparatus

- a) Coors porcelain evaporating dishes (maximum capacity 60 ml).
- b) Reeve Angel 934AH glass fibre discs - either 9.0 or 4.95 cm in diameter.
- c) Buchner funnels of appropriate diameter for filter papers and vacuum filtration unit.
- d) 50 ml wide-mouth volumetric pipets.
- e) Receiving flasks (for filtrate).
- f) Balance - The selected balance must be capable of weighing to five decimal places; a 5-place Mettler H54 and 5-place Sartorius balance are currently used in the River and Sewage laboratories respectively. An electrobalance system is also available in the Sewage laboratory.

Electrobalance system consists of the following modules: 5-place Mettler electrobalance HE 20, digital balance display - Mettler BA28, balance control - Mettler BE20, computer - Hewlett Packard 9810A, typewriter interface - Hewlett Packard 11201A, typewriter - Facit. The computer program is stored on a tape cassette, and listings with other pertinent documentation are filed in the Program Record Book (available from M.W. Rawlings).

Among the balances, the electrobalance system is preferred as it improves test precision, reduces errors associated with record keeping and calculation, reduces paper storage requirements, and decreases somewhat the tediousness of multiple weighings.

4. Reagents

None.

5. Procedure

Dissolved and suspended solids are frequently determined concurrently to economize on the technician's time and equipment. The following description, however, only pertains to dissolved solids determinations.

- a) Dry clean numbered Coors evaporating dishes in oven at $103 \pm 2^{\circ}\text{C}$. Cool and store in a desiccator.
 - b) Dust balance interior; level and zero balance. Weigh Coors dishes designated A and B; ensure that the A plus B and A minus B values conform to specified limits.
 - c) Tare dishes; record weights and dish numbers.
 - d) Filter at least 75 ml sample into a clean receiving flask using pre-washed filter papers and suitable vacuum filtration unit.
 - e) Pipet 50 ml aliquot of filtrate with wide-mouth volumetric pipet into tared evaporating dish. Evaporate overnight in oven maintained at $103 \pm 2^{\circ}\text{C}$; cool and store in a desiccator.
 - f) Dust, level, and zero balance; repeat A & B quality control procedure.
 - g) Weigh evaporating dish plus residue, and record weights. The latter is unnecessary for the electro-balance system; only the order of dishes must be maintained.
- N.B. Analyze at least one out of every 24 samples in duplicate.
- h) Maintenance
 - i) Make certain that the glassware and evaporating dishes are clean.
 - ii) Evaporating dishes must be discarded as soon as they become chipped or pitted.

iii) Keep balance clean at all times.

Follow maintenance procedure outlined in manufacturer's manual, and keep a record of major overhauls, breakdowns, and "remarks".

iv) Check balance accuracy every month with standard weights.

6. Calculations and Reporting

$$\text{Dissolved Solids (mg/l)} = (C - D) \times \frac{10^6}{50}$$

Where *C* is the weight (g) of the Coors dish plus residue.

D is the weight (g) of the Coors dish.

Reporting: Rivers and Lakes - to the nearest mg/l.
Sewage - round off to 5 mg/l.

7. Precision and Accuracy

The accuracy of dissolved solids determination cannot be estimated as the value obtained depends upon the selected experimental conditions. Precision is estimated by analyzing samples in duplicate:

<u>Dissolved Solids Range (mg/l)</u>	<u>Sample Source</u>	<u># Samples</u>	<u>Standard Deviation (mg/l)</u>
0 - 350	River & Lakes	73	3.94
350 - 500	River & Lakes	75	4.36
500 - 750	River & Lakes	38	4.57
0 - 500	Sewage & Trades	12	12.7
500 - 1000	Sewage & Trades	25	22.2
>1000	Sewage & Trades	12	31.5

8. Bibliography

- i) *Standard Methods for the Examination of Water and Wastewater*, 14th ed. p89, APHA, Washington, D.C. 1975.
- ii) *Outlines of Analytical Methods*, Ministry of the Environment, Feb. 25, 1975.

DISSOLVED SOLIDS
CALCULATED FROM CONDUCTIVITY - METHOD B

SUMMARY

Substance determined.	Dissolved solids are calculated from conductivity measurement.
Interpretation of results.	The calculated value (mg/l) assumes that the dissolved solids are ionic species and the sample was collected from a source relatively free of man-made pollution.
Principle of method.	For Ontario lakes and rivers free of industrial pollution, the dissolved solids concentration (mg/l) is 0.65 ± 0.10 times the conductivity ($micromhos/cm$) provided the latter is less than $450 micromhos/cm$ and the pH of the sample lies between 5 and 9 units.
Time required for analysis.	5 minutes.
Range of application	Ontario surface waters relatively free of pollution with conductivities less than $450 micromhos/cm$ and pH values between 5 and 9 units.
Standard deviation.	$2.5 mg/l$ for dissolved solids range 200 - 500 mg/l
Accuracy.	15.5% two-thirds of the time.
Limit of detection.	$3.29s = 8.2 mg/l$.
Interferences and shortcomings.	Conversion factor (0.65 ± 0.10) for conductivity to dissolved solids was developed from a large mass of data of rivers and lakes in Ontario.
Minimum volume of sample.	75 mL.
Preservation and sample container.	Glass bottles should be used and no preservatives may be added.

DISSOLVED SOLIDS
SUMMATION OF IONIC SPECIES - METHOD C

SUMMARY	
Substance determined.	Dissolved solids are calculated from the measured values of 10 parameters.
Interpretation of results.	The calculated value assumes that all the dissolved solids arise from these ten constituents.
Principle of method.	The chemical composition of most surface waters which are relatively free of pollution arises from <i>Ca, Mg, K, Na, H, NH₄, Cl, NO₃, SO₄, and HCO₃</i> ; on drying, the latter would be converted to <i>CO₃</i> . The alkalinity and pH measurements are converted to <i>mg/l</i> of <i>CO₃</i> and <i>H⁺</i> ; the dissolved solids (<i>mg/l</i>) are then estimated by summation. This value is not reported per se, but is used as a quality control check on sample analyses.
Time required for analysis.	5 minutes.
Range of application.	At least 7 of the foregoing parameters are required: <i>Ca, Mg, Na, K, Cl, SO₄</i> , and alkalinity.
Standard deviation.	Not available.
Accuracy.	Not available.
Limit of detection.	Not available.

